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N 65-33127

GPO PRICE $ _____

CSFTI PRICE(S) $ _____

(ACCESSION NUMBER)

_48_
(PAGES)

_CR 64592_
(NASA CR OR TMX OR AD NUMBER)

_____ (THRU)

_1_
(CODE)

_03_
(CATEGORY)

Hard copy (HC) _2.00_

Microfiche (MF) _.50_

ff 653 July 65

INVESTIGATIONS LEADING TO THE DEVELOPMENT
OF A PRIMARY ZINC-SILVER OXIDE BATTERY
OF IMPROVED PERFORMANCE CHARACTERISTICS
6TH QUARTER
Contract No. NAS-8-5493
Control Number TP3-83728
CPB 13-1600-63
George C. Marshall Space Flight Center
Huntsville, Alabama

TABLE OF CONTENTS (Con't)

I. PURPOSE

The immediate objective during this extended contract period shall be the design of a zinc-silver oxide cell capable of activated stand periods and recharge abilities as follows:

    A. Stand period (or useful life) - thirty days
    B. Stand temperature - 90° F
    C. Cycle capability - six cycles in thirty days
       1. Five cycles removing 25% depth
       2. A final discharge of 100% capacity
    D. Battery voltage during discharge - 28 ± 2.0 volts
       (1.40 ± 0.10 volts per cell)

Related studies will be carried out as required to achieve this goal.

II. ABSTRACT

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Studies are being directed towards optimizing electroformation processes for the silver oxide electrode. Preliminary data have confirmed that the use of relatively high silver densities (90-110 gram/in^3) is not a suitable means of increasing the capacity of a cell. Other studies have yielded data relative to the effect of apparent density and discharge rate upon the efficiency of the spongy zinc plate. Other areas of study underway are as follows:

     A. Electrolyte additives
     B. Effects of carbonation upon KOH electrolyte
     C. Correlation of cell impedance to state-of-charge
     D. Methods of recharge
     E. Effects of cell tightness and volume of electrolyte
        upon cell performance.

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III. FACTUAL DATA AND DISCUSSION

A. Electroformation of the Silver Positive Plate

1. Change Efficiency Study

a. Purpose

As zinc-silver oxide batteries are commonly constructed such that
the silver plate limits cell or battery capacity, it is obvious
that any process which results in increased state of oxidation
of the dry-charged silver plate represents a potential increase
in battery capacity. The state of oxidation of the production
positive plate is ordinarily estimated by determining the weight
increase during the electroformation process, although T. Dirkse
(1) has stated that under certain conditions oxygen may be ad-
sorbed by the positive plate during electroformation processes.
Oxygen that is adsorbed in this manner may be released in the
gaseous state upon discharge of the plate without a corresponding
yield of ampere hour capacity. It is believed, however, that
under the constant current method of charge the quantity of oxygen
added to the silver plate is an adequate indication of the state
of oxidation. The object of the studies described below and those
to follow is the investigation of various methods of electro-
formation and, if possible, the improvement of the efficiency
and extent of the electroformation process.

b. Procedure

All plates used in these studies were sintered silver and were
2 7/8 x 6 inches. Expanded silver of 4/0 mesh served as the grid.
Two 20 ga. silver wires were attached to each plate for current
leads. Plate weights in the various tables include only the
weight of the silver sinter. Charge effectiveness studies have
been carried forward in three series as described below.

1. Series

This section involved the utilization of cells consisting
of one silver plate, Vexar separator, (1/4 x 1/4" mesh poly-
propylene), nickel sheet anodes, and 1.300 specific gravity
potassium hydroxide solution as electrolyte. Individual
cells were mounted in plastic cases. Series I cells were
numbered 1-6. Table No. 1 lists the initial silver weight of
each cell. A charge current of 0.087 amps/in^2 (three amps
total) was impressed upon each series-connected cell for the
length of time indicated by Table No. 1. At the end of the
charge time, the plate was removed from the cell, washed
overnight in flowing water, tested with phenolphthalein in-
dicator to confirm the removal of KOH, and dried to constant
weight in a gravity-convection thermostatically controlled
hot-air oven at 165° F. The weight increase was taken to be
due to oxidation of silver. The percent weight increase
based upon sintered silver originally present was calculated.

(1) Dirkse, T.P., Journal of The Electrochemical Society, Vo.117, No. 11, Pg. 862.

An increase of 14.8% would correspond to complete conversion to divalent silver.

2. Series II

Plate and cell construction details for cells of this series are the same as for Series I. All cells of Series II (Cell Nos. 7-12) were charged at a three ampere rate for forty-five hours, then discharged at six amperes for five and one-half hours. Cells were then again placed on charge at three amperes. After 70.5 hours, the silver plate in Cell No. 7 was removed for weight increase evaluation. Cell Nos. 8 and 9 were removed following total times of 83 and 95.5 hours respectively. At the end of 95.5 hours, Cell Nos. 10 and 11 were placed on charge at the six ampere rate. These cells were removed following four and eight hours at the increased charge rate. (99.5 and 103.5 hours total respectively). The plate in Cell No. 12 was removed at the end of 117 hours, after having been on charge at 3 amperes continuously from the 50.5 hour point with no high rate-of-charge boost. Rinsing, drying, and weight increase determinations were the same as for Series I.

3. Series III (Cell Nos. 13-17)

Cell construction techniques again were the same as for Series I. A cells were placed on charge at the three ampere rate for 45 hours. For the following eleven hours (to t=56) cells were placed on discharge at a six hour rate. Cells were returned to the three ampere charge rate following 56 total hours. After 83 hours, Cell No. 13 was disassembled and the positive plate rinsed, dried and weighed. The plate from Cell No. 14 was removed at the end of 95.5 hours. Cell Nos. 15 and 16 were placed on charge at six amperes at t=95.5 hours. Cell Nos. 15 and 16 were removed following 99.5 hours and 103.5 hours total and treated as all other plates. Cell No. 17 was removed after a total of 117 hours, having charged continuously at three amperes from t= 56 hours. Weight increases for cells of Series III are listed by Table No. 1(b).

c. Results and Discussion

Charge Effectiveness Investigation - The results and conditions of formation of silver plates under various procedures are tabulated in Table Nos. 1-16. A summary of the results is presented by Table No. 2. The silver plate from Cell No. 4 displayed the largest percent weight gain of the 17 plates in this group. This was 13.35% (90% of theoretical). This plate was subjected to 83 hours uninterrupted charge at the three ampere rate. Table No. 1a indicates that there may be considerable plate-to-plate variation in charge, characteristics after the gassing potential is reached. There was no obvious consistent variation of plate weight increase with additional charge, nor was there any confirmation of significant increase in weight after 83 hours continuous charge at the three ampere rate. Table No. 2 lists

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plates from Series Nos. I-III which displayed the greatest weight increases, along with their charge procedures.

2. **Fractional Factorial Plan Experiment**

   a. **Purpose**

   In order to investigate possible effects of electroformation current density, temperature, plate weight and apparent density upon the electroformation process, the following test was conducted. A fractional factorial experiment was designed following Addelman's Plan No. 11 (using columns 6, 7, 8 and 9) found in "Orthogonal Main-Effect Plans" Addelman-Kempthorne (Iowa State University). The plan was followed utilizing twenty-seven trials (3^{4-1}); a 1/3 full factorial. The factors and levels were:

FACTORS	LEVELS		
	High	Medium	Low
Temperature (F)	120	100	80
Current density (amp/in^2)	0.088	0.044	0.022
Silver ewight (gm/in^2)	9.5	9.0	8.5
Silver density (gm/in^3)	110	100	90

   b. **Procedure**

   Details of the plan are listed in Table No. 4, in which the combinations of levels incorporated into each test cell are delineated. Plate dimensions, grid and terminals for these test cells were the same as previously described. However, the individual plates were carefully selected to conform to the levels of silver weight and apparent density. Approximately 250 ampere hours charge were impressed upon each plate during the formation process. In terms of elasped time of formation this means that the nine plates under the high current density charge were formed for approximately 84 hours; those under medium current density for approximately 166 hours; those under the low current density charg were formed for approximately 332 hours. At the end of the appropriate charge time, the plates were removed, washed, dried, and weighed as described previously. The percent weight gain was considered to be "response" of the plates and these data were utilized in the statistical analyses given in the following sections.

   The following methods of analysis were performed utilizing the responses mentioned: 1) Mathematics for half-normal plot (no plot made, since only four comparisons were studied); 2) Analysis

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by comparison of means gave fairly conclusive indications, the other analyses were conducted to allow estimation of the "significance" of the effects of the test variables.

The response values for the individual cells (plates) as accumulated in this experiment are tabulated in Tables No. 3 and No. 4. As indicated previously, these were the calculated percents of increase in weight for the silver in the plates. Table No. 4 shows the plan in detail with the levels of each factor for the individual plates given in detail. The level of each factor is shown as follows: low = 0; medium = 1; high = 2. Also shown in Table No. 4 are the coded values of the levels arranged for the computation of the linear comparison of each effect. These are: 0, -1, 1, (as shown). This analysis yielded the significance of effects values displayed in Table No. 5. It appeared, therefore, that the linear comparison of density of Ag in the plates was the notably significant effect. It also appeared that the significance lay in the direction of low density which means that lower density yields larger responses. There is a certain amount of subjective interpretation involved, however, since no normal plot of significance was possible. This was due to the fact that only four comparisons of effects were made. To construct a complete half-normal plot twenty-six values are required, since there are twenty-six degrees of freedom available.

In view of the above, an additional analysis was performed in which the data were analyzed by a comparison of the means of the responses accumulated. In Table Nos. 6 through 8 are listed the responses arranged for this analysis. It should be pointed out that data are displayed to show the effects of temperature, silver weight, and silver density as collected at the three rates of current density, high, medium and low. The level of each effect is shown as follows: high = H, medium = M, low = L. The detailed analysis of the comparison of means are displayed in the following section.

c. Analysis of Experimental Results

1. High Current Density (0.088 amp/in^2)

a. Temperature

Low	Medium	High	Mean
9.51	10.44	9.42	9.79

Formation at the medium temperature (100° F) yielded a gain of 0.65 units higher than the mean.

b. Silver Weight (grams)

Low	Medium	High	Mean
9.99	9.82	9.60	9.80

Formation at the medium weight of silver yielded a gain of 0.19 units higher than the mean.

c. Silver Density $(grams/in^3)$

Low	Medium	High	Mean
12.19	9.79	7.46	9.81

Formation at the low silver density yielded a gain 2.28 units higher than the mean.

The optimum conditions at high current density appeared to be: Medium temperature, low silver weight and silver density.

2. Medium Current Density $(0.044 \ amp/in^2)$

a. Temperature

Low	Medium	High	Mean
11.47	9.42	9.84	10.24

Formation at the low temperature yielded a gain 1.23 units higher than the mean.

b. Silver Weight (grams)

Low	Medium	High	Mean
10.02	10.63	10.09	10.24

Formation at the medium silver weight yielded a gain 0.19 units higher than the mean.

c. Silver Density $(grams/in^3)$

Low	Medium	High	Mean
11.63	10.66	8.78	10.36

Formation at the low silver density yielded a gain 1.27 higher than the mean.

The optimum conditions at medium current density appeared to be: Low temperature, medium weight and low density.

3. **Low Current Density** (0.022 amp/in^2)

    a. **Temperature**

Low	Medium	High	Mean
10.77	9.54	9.24	9.85

    Formation at the low temperature yielded a gain 0.92 units higher than the mean.

    b. **Silver Weight** (grams)

Low	Medium	High	Mean
10.34	11.11	9.16	10.20

    Formation at the low silver weight yielded a gain 0.91 units higher than the mean.

    c. **Silver Density** (grams/in^3)

Low	Medium	High	Mean
10.87	9.27	9.39	9.84

    Formation at the low silver density yielded a gain 1.03 units higher than the mean.

    The optimum conditions at low current density appeared to be: low temperature, low silver weight, and low silver density.

The above analysis also indicated that the preferred effect (common to all current densities used) was that of low silver density.

In order to confirm this as the significant effect it was decided to analyze the data by the statistical method, requiring considerable mathematical calculation of Analysis of Variance. Accordingly, the data were arranged for the Analysis Variance. This arrangement appears in Table No. 9, in which the data are arranged for the determination of the effect of temperature; Table No. 10, in which the data are arranged for the determinatio of the effect of current density; Table No. 11, in which the data are arranged for the determination of the effect of silver weight and Table No. 12, in which the data are arranged for the determination of the effect of silver density. The detailed computation is shown at the bottom of each of the tables mentioned above. Each of these required the calculation of a correction factor, total sum of squares, and a sum of squares for the effect being analyzed.

These values are summarized in Table No. 13, a summary of the analysis of variance. The total degrees of freedom are 26, since there were 27 samples (trials). Each effect investigated has 2 degrees of freedom, since there were three levels of each effect involved. Therefore, the residual (error) degrees of freedom are 18, by difference. The corrected sum of squares for each effect (Table Nos. 9 through 12) was divided by the number of degrees of freedom to obtain a "mean squares" value for each. Each mean squares value was then divided by the residual (error) mean squares figure to yield the F test value. These F test values when compared to those appearing in Davies "Design and Analysis of Industrial Experiments" yielded the significance values shown. The only effect shown to be significant by this Analysis of Variance was that of the silver density.

In order to determine which level (high, medium, low,) of silver density was preferred the calculation shown in Table No. 14 was carried out. A computation of variance for each level was made and these were compared with the mean response for each level. For high silver density the variance was found to be 21.53 compared with a mean of 8.41. For medium silver density the variance was found to be 18.88 compared with a mean of 9.91. For low silver density the variance was found to be 16.23 compared with a mean of 11.57. In this analysis the low density silver was again determined to be preferred.

B. Electrolyte Additives

1. Object

The objective of this area of study is to evaluate electrolyte additives which might cause "gelling" and therefore, retention of electrolyte within the separator system.

2. Procedures

As a preliminary step attempts were made to prepare solutions of two materials in potassium hydroxide - water systems. These were carboxymethyl hydroxyethyl cellulose and a commercial product designated as Vee Glass. The latter is essentially a mixture of mineral silicates and oxides, which, upon dispersions of one, two and four per cent (by weight) of Vee Glass in 1.400 specific gravity hydroxide solution, and two, four and six per cent CMHEC in 1.400 specific gravity potassium hydroxide solution. This was done by measuring the appropriate ratios of materials and mixing for three minutes at high speed using a Waring Blender.

3. Results

CMHEC dispersed readily in the electrolyte solution. Upon activation of cells with these gels, the liquid portion was absorbed by the cell components, leaving a residue of solids above the cell groups.

The Vee Glass apparently did not disperse in the caustic solution, as indicated by the settling of material and the apparent lack of increase in the viscosity of the electrolyte. Other products will be examined for the desired dispersion properties and evaluated in cells if preliminary tests are promising.

C. Effect of Carbonation of Electrolyte

1. Object

In view of the degradation of cellulosic separator membranes, with the resulting carbonation of the electrolyte, it was considered worthwhile to evaluate the effects of progressive carbonation upon the pH and conductivity of a 1.400 specific gravity solution of potassium hydroxide.

2. Procedure

Dry CO_2 gas was bubbled into a caustic solution at a measured rate of flow. Both conductivity and pH were constantly monitored. Samples of electrolyte were taken periodically for analysis of potassium hydroxide and potassium carbonate content.

3. Results

This determination has been completed. However, certain temperature corrections upon the conductivity values must be computed prior to the final correlations. These data will be reported in the monthly report covering progress through January, 1965.

D. Cell Capacity and Effects of Top Charge

Orders have been placed with the Eagle-Picher Department for the cells necessary for the following determinations.

1. A statistical estimate of cell capacity as constructed, that is, with no charging prior to initial discharges.

2. Estimation of the charge acceptance of freshly activated cells at an 0.5 ampere rate to an end-of-charge voltage of 1.98 volts per cell.

3. Initial discharge capacity of cells top-charged as in "2" above. Difficulty in production scheduling has prevented the delivery of these cells to the present time.

E. Effect of Cell Tightness and Quantity of Electrolyte

A study is planned to evaluate the combined effects of cell tightness and quantity of electrolyte in the cell. This will be done by employing three volumes of electrolyte in test cells at each of three levels of tightness. This study will be performed as a full replicate. That is, all possible combinations will be tested. The following is a summary of pertinent factors of the study:

Factor A, Cell Tolerance
A_0 - 0.025 inch tolerance
A_1 - 0.000 inch tolerance
A_2 - -0.025 inch tolerance

Factor B, Volume of Electrolyte
B_0 - 19.0 cc electrolyte per cell
B_1 - 19.5 cc per cell
B_2 - 20.0 cc per cell

The cell tolerance is that difference between the internal thickness of the cell case and the calculated total thickness of wet cell components. Level A_2, which requires a negative tolerance, will be obtained by using plates of such thickness that the cellulosic separator will not be allowed to swell to its maximum ability.

Ratios of positive to negative material in the cells will be kept constant. This means that cell theoretical capacity will vary with the designed tolerance. For this reason, efficiency of utilization of active material will be used as a response, rather than cell discharge capacity.

F. Effect of Activated Stand Upon Cell Impedance

1. Object

It is hoped that these studies might lead to a procedure for determining cell state-of-charge or the presence of defective cells.

2. Procedure

Immediately following activation, cells were placed on open circuit stand at +90° F. Open circuit voltages were monitored daily. In addition, cell impedance was measured periodically by passing 0.5 amperes sinusoidal current through the cells and measurnng the resultant A.C. voltage drop across the cells. Figure No. 1 reveals that impedance of freshly activated cells is quite small and does not indicate a point of resonance within the range of frequencies used. Figure No. 2 exhibits the effect of open circuit stand upon cell impedance measured at 500 cps. Figure Nos. 3 and 4 reveal the effect of frequency of determination upon the measured cell impedance following thirty days activated stand. These figures relate to cells having open circuit voltages of 1.85 and 1.60 volts, respectively.

3. Results

It can be seen that the cell which retained a 1.85 open circuit voltage exhibited greatly increased impedance, and no condition of resonance was noted at frequencies up to 15 K cps. The cell having an open circuit voltage of 1.60, however, displayed very low impedance and exhibited a pronounced minimum impedance in the area of 500-1000 cps. Following a discharge, all cells exhibited impedances of the magnitude and frequency disclosed in Figure No. 4. This indicates that cell impedance is a function of the quantity of metallic silver in the positive plate. Cells apparently display low impedance immediately following activation because some metallic silver exists in the dry-charged silver plate. Upon wetting of the plate with electrolyte, local action within the positive plate results in the reaction between metallic silver and divalent silver to produce monovalent silver oxide. This depletion of metallic silver within the plate and resultant formation of monovalent silver oxide (which is more resistant than the divalent oxide) apparently causes the increased cell impedance.

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G. Full-Cell Evaluation - Separation Variables

A number of separator combinations have been evaluated in full sized test cells. These were cells having a mean actual capacity of approximately 13.0 ampere hours at the thirty ampere discharge rate. These cells were constructed to provide data relative to the effect of various separator systems upon cell performance.

1. Cycle Evaluation

Combinations of separator materials employed in a preliminary cycle capability studies are listed in Table No. 15. All cells, except where otherwise noted, contained two wraps of 300 Cellophane between positive and negative plates. All cells contained approximately thirty-eight grams each silver and zinc.

TABLE NO. 15
Separator Evaluation - Preliminary Design Study

CELL TYPE	1 & 5	2	3 & 4	6 & 7	8 & 9
Positive "open" separator	#9526 Nylon	#9526 Nylon	EM-470	EM-312	M-1406
Negative "open" separator	R-35-D	R-35-D	EM-470	EM-312	M-1406
Cycle No. 1 Discharge capacity to 1.10V (ampere hours)	10.5	11.1	8.5	10.1	10.75
Cycle No. 5 Discharge capacity to 1.10V	11.9	*	11.8	11.5	12.0

Material Identification

#9526 Nylon - Woven Polyamide - 0.0035 inches thick
EM-312 - "Entangled" nylon fibers with Dynel binder, - 0.0035 inches thick
M-1406 - "Entangled" nylon fibers with Dynel binder, - 0.0025 inches thick
R-35-D - Non-woven rayon fibers, - 0.0035 inches thick
EM-470 - Non-woven Dynel

* This cell failed to accept recharge following the initial discharge.

2. Test Procedure

Cells were activated with 20 cc of 1.300 specific gravity potassium hydroxide solution and allowed to soak for four hours. They were then discharged at a thirty ampere rate to 1.10 volts per cell. This discharge was conducted to determine the effects of the various open separators upon initial performance characteristics. Although only voltage above 1.30 volts per cell is useful, the discharges were carried further to determine the nature of the "knee" of the discharge curves.

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Cells were then recharged at 2.0 amperes to an end voltage slightly lower than 2.0 volts per cell - the point at which there was visible evidence of the deposition of zinc from the electrolyte. Test cells were then entered into a charged stand at a temperature of $90 \pm 2°$ F. Three discharges of six minutes duration at a rate of thirty amperes were conducted in the following month, after which cells were recharged as described above. At the conclusion of this thirty day period, a full capacity discharge was again conducted. Results of these discharges are exhibited by Figure Nos. 5 through 9.

3. Results

One cell, No. 2, which contained a membrane of irradiated Teflon, failed to accept a charge following the initial discharge. Upon examination, it was found that both silver and zinc had penetrated the separator system resulting in internal shorting. The remaining cells contained two layers of cellophane and were treated as follows:

During the final discharge, considerable heating of the cells was noted, as indicated by plastic deformation of some of the cell cases. As revealed by the discharge curves, the capacity of all cells was greater following the thirty day period. This results from the fact that cells were charged to a higher state-of-charge than existed at the time of cell construction. This indicates the possible advantages of a "boost" charge immediately following activation and preceeding the initial discharge. It is also possible that degradation of the cellulosic membrane lowered its electrical resistance, resulting in sustained voltage at a higher level.

Cells ere "post-mortemed" and examined for evidence of near-failures. It was found that silver had penetrated to the second layer of cellophane, although the layer closest to the positive plate contained approximately 4.25 times as much silver. Average total silver loss per cell was 1.64 grams, or about 5% of the total. Silver penetration of this magnitude is regarded as an indication that in a larger sample size some failures might be expected.

H. Negative Material Formulation.

1. Objective

As reported previously, the capacity efficiency of the zinc plate is increased within limits by the use of less dense material. Attention has been given to the combined effects of zinc apparent density and discharge current density upon the efficiency of zinc. These data are necessary to determine the ratio of positive and negative materials necessary for optimum performance.

2. Procedure

Zinc plates of the size employed in test cells were used to construct negative-limited cells. That is, care was taken to insure an excess of positive active materials. All negative plates contained 1.45 grams zinc per square inch, but were pressed to varying thicknesses to obtain the desired material densities. These test cells were then discharged at varying current densities, and the re-

sulting capacity efficiencies calculated. Table No. 16 summarizes the pertinent data. Figure No. 14 reveals the effects of zinc apparent density and discharge current density.

3. Conclusions

It is apparent that for plates of the weight investigated efficiency increases with decreasing material density. As suggested previously, capacity advantages on a volume basis diminish with very low densities of 35-40 grams per cubic inch. At densities in excess of 44 grams per cubic inch, however, the volumetric energy density decreases rapidly, as indicated by the fifth column of Table No. 16.

These data were obtained on initial discharges, and do not reflect possible effects of decreased apparent density upon erosion during stand.

Interpolation of the data in Table No. 16 indicates a possible zinc capacity of just over thirteen ampere hours at the thirty ampere discharge rate for the full size test cells previously described. The fact that this capacity was not realized in these cells indicates that they may have been positive limited. It is planned to obtain similar efficiency data for the silver oxide plate to ensure a more equal practical capacity balance.

TABLE NO. 16
Effects of Apparent Density of Zinc
Upon Discharge Efficiency
(At rate of 0.58 amp/in^2)

Apparent Density (Gm/in^3)	Thickness (inches)	Efficiency (Per Cent)	Capacity (A. H.)	Capacity (A.H./milli-inch Thickness)
35	0.042	51	1.98	0.047
39	0.038	45	1.75	0.046
44	0.034	38	1.47	0.043
50	0.030	30	1.16	0.039
58	0.026	21	0.81	0.031

IV. SUMMARY AND CONCLUSIONS

A. Studies are continuing relative to improvement of the electroformation of silver positive plates. Preliminary studies have indicated that the effect of silver apparent density upon electroformation efficiency overshadows the effects of temperature variations of 80-100° F, formation current densities of 0.022-0.088 amperes/square inch, and plate weights of 8.5-9.5 grams/square inch.

Silver plates having apparent densities of 90-100 grams/in^3 were found to achieve states of oxidation of only approximately 90% of the theoretical. Apparent densities of 90 gm/in^3 resulted in more effective electroformation than higher densities, the 90 gm/in^3 plates achieving 10-15 per cent greater state-of-charge than 110 gm/in^3 plates for a corresponding duration of charge.

Additional studies of densities of 70-95 grams/in^3 are being conducted.

B. Electrolyte Additives

No favorable results have been obtained in efforts to produce stable gel electrolytes. A commercial product designated as Vee-Glass refused to disperse in KOH solutions. Carboxymethyl hydroxyethyl cellulose (CMHEC) produced gels which were broken down upon cell activation.

C. Data relative to the effects of progressive carbonation of 1.400 specific gravity electrolyte are being tabulated. These data were obtained by reacting dry CO_2 gas with electrolyte, while monitoring electrical conductivity and pH.

D. Further full-cell separation evaluations indicate that several separator systems yield comparable initial discharge characteristics, with no apparent effects on cell capacity and minor effects upon voltage.

E. Cell impedances determined by A-C methods apparently vary characteristically with the frequency employed in the determination. Effort will be made to determine whether it is possible to determine cell state-of-charge by measuring cell impedance.

V. PROGRAM FOR THE NEXT INTERVAL

The following phases of study are planned for the following quarter:

A. Continuation of positive electroformation. Various silver plate densities ranging from 70-90 gram/in^3 will be studied relative to speed and efficiency of electroformation.

B. Cells employing three ratios of positive-to-negative active material will be tested to determine whether the present ratios are near the optimum for the applicable rate of discharge.

C. Studies will be begun, on a statistical evaluation basis, relative to the combined effects of all tightness and volume of electrolyte.

D. Recharge and "top-charge" techniques will be studied, with the object of maintaining maximum capacity on cycle.

E. The relationship between cell impedance and state-of-charge will be further examined.

VI. PERSONNEL

The following number of man-hours have been expended during the contract period:

Engineering	-	3213 hours
Technical	-	5826 hours
TOTAL		9039 hours

A P P E N D I X

TABLE NO. 1

DISCHARGE-CHARGE EFFECTIVENESS
SILVER PLATE FORMATION STUDY
Series I (formed at 0.087 amps/in^2)

Cell No.	Ag Initial Wt. (gms)	Ag Final Wt (gms)	Wt. Gain	Chg. Hrs.	% Wt Gain
1	130.36	146.13	15.77	30	12.10
2	128.60	144.17	15.57	45	12.11
3	129.01	144.49	15.48	59	11.99
4	130.75	148.20	17.45	83	13.35
5	129.11	145.90	16.79	95.5	13.00
6	128.62	144.55	15.93	117	12.39

TABLE NO. 1a

DISCHARGE-CHARGE EFFECTIVENESS
SILVER PLATE FORMATION STUDY
Series II (formed as noted)

Cell No.	Ag Initial Wt. (gms)	Ag Final Wt (gms)	Wt Gain	Chg. Hrs.	% Wt Gain
7	125.58	141.86	16.28	70.5	10.96
8	137.55	144.05	16.50	83.0	12.94
9	130.19	145.94	15.75	95.5	12.99
10	126.25	144.35	16.10	99.5	12.55
11	128.20	144.55	16.35	103.5	12.75
12	130.55	147.55	16.70	117.0	12.76*

All plates formed at 0.097 amps/in^2 first 45 hours; all
discharged at 0.174 amps/in^2 45 - 50.5 hours; all on 0.087
amps/in^2 charged at 50.5 hours.

* Cell No. 12 charged at 3 amps continuously from t=50.5 hours

TABLE NO. 1b

DISCHARGE-CHARGE EFFECTIVENESS
SILVER PLATE FORMATION STUDY
Series III (formed as noted)

Cell No.	Ag Init. Wt. (gms)	Ag Final Wt. (gm.)	Wt. Gain (gm.)	Charge Hrs.	% Wt. Gain	
13	130.35	147.30	16.95	93	13.00	
14	129.05	145.35	16.30	95.5	12.63	
15	124.71	143.35	18.64	99.5	12.83	(1)
16	128.35	144.60	16.25	103.5	12.66	(2)
17	131.40	148.14	16.74	117.0	1.61	(3)

All plates formed at 0.087 amp/in^2 first 45 hours; all on
0.174 amps/in^2 discharge 45 to 56 hours; at 56 hours placed
on 0.087 amps/in^2 charge.

(1) 0.174 amp/in^2 charge 95.5 - 99.5 hours

(2) 0.174 amp/in^2 charge 95.5 - 103.5 hours

(3) 0.087 amp/in^2 charge 56 - 117 hours

TABLE NO. 2

COMPARISON OF % WEIGHT GAINED

Hours	SERIES I Cell No.	% Wt Gain	SERIES II Cell No.	% Wt Gain	SERIES III Cell No.	% Wt Gain
30	1	13.10				
45	2	12.17				
50	3	11.91				
70.5			7	12.76 (4)		
83	4	13.35 (1)	8	12.94 (5)	13	13.00
95.5	5	13.00 (2)	9	12.13 (3)	14	12.63
99.5			10	12.55	15	12.83
103.5			11	12.75	16	12.66
117.0	6	12.39	12	12.76	17	12.61

NOTES:

1. #4 cell plate charged 83 hours at 0.087 amp/in^2 rate.

2. #5 cell plate charged 95.5 hours at 0.087 amp/in^2 rate.

   #13 cell plate charged 45 hours at 0.087 amp/in^2 rate;
   11 hours discharged 0.174 amp/in^2 rate; 17 hours charged
   at 0.087 amp/in^2 rate.

3. #9 cell plate charged 45 hours at 0.087 amp/in^2 rate; dis-
   charged 5.5 hours at 0.174 amp/in^2 rate; charged 20 hours
   at 0.087 amp/in^2 rate.

4. #7 cell plate charged 45 hours at 0.087 amp/in^2 rate;
   discharged 5.5 hours at 0.174 amp.in^2 rate; charged
   20 hours at 0.087 amp/in^2 rate.

5. #8 cell plate charged 45 hours at 0.087 amp/in^2 rate; dis-
   charged 5.5 hours at 1.174 amp/in^2 rate; charged 33 hours
   at 0.087 amp/in^2 rate.

SILVER PLATE FORMATION STUDY
RESPONSES FOR FACTORIAL EXPERIMENT

Cell No.	Initial Ag Weight	Final Ag Weight	% * Wt Gain
1	132.75	150.25	13.19
2	141.35	153.13	8.33
3	148.17	162.65	9.77
4	139.10	154.85	11.32
5	143.00	161.66	13.04
6	134.70	145.71	8.17
7	152.26	163.45	7.80
8	133.70	147.00	9.97
9	138.39	154.41	11.58
10	132.95	146.56	10.26
11	141.85	158.85	12.00
12	147.90	162.05	9.58
13	138.75	153.38	10.58
14	143.63	157.15	9.44
15	134.26	144.45	7.68
16	154.04	164.20	7.44
17	132.90	141.25	6.28
18	138.66	154.22	11.29
19	132.85	149.07	12.24
20	141.95	155.75	9.73
21	151.17	166.65	10.23
22	138.90	152.58	9.84
23	143.56	162.31	13.05
24	134.10	145.35	8.39
25	153.45	162.95	6.18
26	133.89	149.47	11.65
27	138.65	152.51	10.00

* Used as response figure in all subsequent calculations.

TABLE NO. 4

PLAN (FACTORIAL) FOR FORMATION STUDY

	Factor Levels				Analysis (half normal plot)				
TEST	TEMP	C.D.	WT.	DENS.	RESPONSE	T_L	C_L	WI_L	DEN_L
1	0	0	0	0	13.2	0	0	0	0
2	1	2	1	2	8.3	-1	1	-1	1
3	2	1	2	1	9.8	1	-1	1	-1
4	0	0	1	1	11.3	0	0	-1	-1
5	1	2	2	0	13.0	-1	1	1	0
6	2	1	0	2	8.2	1	-1	0	1
7	0	0	2	2	7.8	0	0	1	1
8	1	2	0	1	10.0	-1	1	0	-1
9	2	1	1	0	11.6	1	-1	-1	0
10	1	1	0	0	10.3	-1	-1	0	0
11	2	0	1	2	12.0	1	0	-1	1
12	0	2	2	1	9.6	0	1	1	-1
13	1	1	1	1	10.6	-1	-1	-1	-1
14	2	0	2	0	9.4	1	0	1	0
15	0	2	0	2	7.7	0	1	0	1
16	1	1	2	2	7.4	-1	-1	1	1
17	2	0	0	1	6.3	1	0	0	-1
18	0	2	1	0	11.3	0	1	-1	0
19	2	2	0	0	12.2	1	1	0	0
20	0	1	1	2	9.7	0	-1	-1	1
21	1	0	2	1	10.2	-1	0	1	-1
22	2	2	1	1	9.8	1	1	-1	-1
23	0	1	2	0	13.1	0	-1	1	0
24	1	0	0	2	8.4	-1	0	0	1
25	2	2	2	2	6.2	1	1	1	1
26	0	1	0	1	11.7	0	-1	0	-1
27	1	0	1	0	10.0	-1	0	-1	0

Effect*	$\Sigma c X_i$	$\Sigma c X_i / \Sigma c$	Significance
T_L	-4.2	0.993	0.10
CD_L	-4.3	-1.01	0.10
Wt_L	-1.2	-0.284	0.00
$Dens_L$	-13.6	-5.21	0.01

NOTE: Above significance values obtainable from slope of a half-normal plot.

* Effect

T_L = Temp., linear

CD_L = Current density - linear

Wt_L = Ag weight, linear

$Dens_L$ = Ag density, linear

Analysis by Comparison of Means
(Arranged with Respect to Current Density)

High Current Density (0.087 amp/in²)
A. Temperature

Cell	Temp.	Ag Wt.	Ag Dens.	Response	Mean	Gp. Mean
10	L	L	L	12.24	9.42	
22	M	M	M	9.84		
25	H	H	H	6.18		
2	M	M	H	8.22		
5	M	H	L	13.04	10.14	9.79
8	M	L	M	9.97		
12	L	H	M	9.58		
15	L	L	L	7.58	9.51	
18	L	M	H	11.29		

B. Ag. Weight

Cell	Temp.	Ag Wt.	Ag Dens.	Response	Mean	Gp. Mean
5	M	H	L	13.04		
12	L	H	M	9.58	9.60	
25	H	H	H	6.18		
2	M	M	H	8.33		
18	L	M	L	11.29	9.82	9.60
22	H	M	M	9.84		
8	M	L	H	9.97		
15	L	L	H	7.58	9.09	
10	H	L	L	12.24		

C. Ag. Density

Cell	Temp.	Ag Wt.	Ag Dens.	Response	Mean	Gp. Mean
2	M	M	H	8.33		
15	L	L	H	7.58	7.46	
25	H	H	H	6.18		
8	M	L	M	9.97		
12	L	H	M	9.58	9.79	9.81
22	H	M	M	9.84		
5	M	H	L	13.04		
18	L	M	L	11.29	12.10	
10	H	L	L	12.24		

Overall mean = 9.80

Table No. 7
Silver Plate Formation Study
Analysis by Comparison of Means
Arranged with Respect to Current Density

Medium Current Density (0.04 amp/in^2)
A. Temperature

Cell	Temp.	Ag Wt.	Ag Dens.	Response	Mean	Gp. Mean
3	H	H	M	9.77		
6	H	L	L	8.27	9.87	
9	H	M	H	11.58		
10	M	L	L	10.26		
13	M	M	M	10.58	9.42	10.24
16	M	H	H	8.44		
20	L	H	H	9.73		
23	L	H	L	13.05	11.47	
26	L	L	M	11.65		

B. Ag Weight

3	H	H	A	9.77		
16	M	H	H	7.44	10.09	
23	L	H	L	13.05		
9	H	M	L	11.58		
13	M	M	M	10.58	10.63	10.24
20	L	M	H	9.73		
6	H	L	H	8.17		
10	M	L	L	10 26	10.02	
26	L	L	M	11.65		

C. Ag Density

6	H	L	H	8.17		
16	M	H	H	7.44	8.78	
20	L	M	H	9.73		
3	H	H	M	9.77		
13	M	M	M	10.58	10.50	10.30
26	L	L	M	11.65		
9	H	M	L	11.58		
10	M	L	L	10.26	11.58	
23	L	H	L	13.05		

Overall mean = 10.25

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Table No. 8
Silver Plate Formation Study
Analysis by Comparison of Means
Arranged with Respect to Current Density
Low Current Density (0.022 amp/1 .)

Cell	Temp.	Ag Weight	Ag Dens.	Response	Mean	Gp. Mean
A. Temperature						
11	H	M	H	12.00		
14	H	H	L	9.44	9.24	
17	H	L	M	6.68		
21	M	H	M	10.23		
24	M	L	H	8.39	9.54	9.85
27	M	M	L	10.00		
1	L	L	L	13.19		
4	L	M	M	11.32	10.77	
7	L	H	M	7.80		
B. Ag Weight						
14	H	H	L	9.44		
21	M	H	M	10.23	9.16	
7	L	H	H	7.80		
11	H	M	H	12.00		
27	M	M	L	10.00	11.11	10.20
4	L	M	M	11.32		
17	H	L	M	9.44		
24	M	L	H	8.39	10.44	
1	L	L	L	13.19		
C. Ag Density						
11	H	M	H	12.00		
24	M	L	H	8.39	9.39	
7	L	H	H	7.80		
17	H	L	M	6.28		
21	M	H	M	10.23	9.27	9.84
4	L	M	M	11.32		
14	H	H	L	9.44		
27	M	M	L	10.00	10.87	
1	L	L	L	13.19		

Overall mean = 9.45

Table No. 9
Silver Plate Formation Study
Responses Arranged for Analysis of Variance

Effect of Temperature

	High	Medium	Low	Total
	9.77	8.33	13.19	31.29
	8.17	13.04	11.32	32.53
	11.58	9.97	7.80	29.35
	12.00	10.26	9.58	31.84
	9.44	10.58	7.68	27.70
	6.28	7.44	11.29	25.01
	12.24	10.23	9.73	32.20
	9.84	8.39	13.05	31.28
	5.18	10.00	11.65	27.83
Σx_i	85.50	88.24	95.29	269.03
$(\Sigma x_i)^2$	7310.25	7786.30	9080.18	72,377.14

Correction Factor $= (269.03)^2 \div 27 = 2680.63$

Total Sum of Squares $= (\text{Total, each response})^2 - CF$
$$= 2782 - 2680 = 102$$

Temperature SS $= \dfrac{(7310.25 + 7786.30 + 9080.18)}{9} \quad - CF$

$$= 2686.30 - 2680.63$$

$$= 5.67$$

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Table No. 10
Silver Plate Formation Study
Responses Arranged for Analysis of Variance

Effect of Current Density

	High	Medium	Low	Total
	6.13	9.77	13.19	31.29
	13.04	8.17	11.32	32.53
	6.97	12.58	7.50	29.35
	9.58	19.26	12.49	31.34
	7.68	10.58	9.44	27.70
	11.29	7.44	6.28	25.01
	12.24	9.73	10.23	32.20
	9.84	13.05	8.39	31.28
	6.18	11.55	10.10	27.83
Σx_i	88.15	92.23	88.65	269.03
$(\Sigma x_i)^2$	7770.42	8509.38	7858.82	72,377.14

Correction Factor = $(269.3)^2 \div 27 = 2680.03$

Total Sum of Squares = (Total, each response)^2 - CF
                     = 2782 - 2680 = 102

Current Density SS = $\dfrac{(7770.42 + 8509.38 + 7858.82)}{9}$ - CF

= 2681.13 - 2680.03
= 1.10

Table No. 11
Silver Plate Formation Study
Responses Arranged for Analysis of Variance

Effect of Silver Weight

High	Medium	Low	Total
9.77	8.33	8.19	31.29
13.04	11.32	5.17	32.53
7.80	11.58	9.97	29.35
9.58	12.00	10.26	31.84
9.44	10.58	7.58	27.70
7.15	11.20	5.28	26.01
10.23	9.73	12.24	32.2
13.05	5.84	8.39	31.28
6.15	10.00	11.58	27.83

	High	Medium	Low	Total
ΣX_i	86.63	94.57	87.83	269.03
$(\Sigma X_i)^2$	7487.44	8952.41	7714.11	72,377.16

Correction Factor $= (269.03)^2 \div 27 = 2680.53$

Total Sum of Squares $= (\text{Total, each response})^2 - CF$
$$= 2782 - 2680 = 102$$

Silver Wt. SS $= \dfrac{(7487.44 + 8952.41 + 7714.11)}{9} - CF$

$$= 2684.09 - 2680.53$$
$$= 4.36$$

Table No. 12
Silver Plate Formation Study.
Responses Arranged for Analysis of Variance

Effect of Apparent Density

	High	Medium	Low	Total
	6.33	9.77	15.15	31.20
	8.17	11.32	13.04	32.53
	7.50	9.97	11.88	29.35
	12.00	9.58	10.26	31.84
	7.58	10.68	9.44	27.70
	7.44	6.28	11.29	25.01
	9.73	10.23	12.24	32.20
	8.39	9.84	13.05	31.28
	6.18	11.65	10.00	27.53
Σx_i	75.75	89.22	114.09	269.03
$(\Sigma x_i)^2$	5733.56	7960.21	10,834.73	72,377.14

Correction Factor $= (269.03)^2 \div 27 = 2680.63$

Total Sum of Squares $= ($ total, each response$)^2 - CF$
$$= 2782 - 2680 = 102$$

Apparent Density SS $= \dfrac{(5733.52 + 7960.21 + 10,834.73)}{9} - CF$
$$= 2725.38 - 2680.63$$
$$= 44.75$$

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Table No. 13
Silver Plate Formation Study

SUMMARY OF ANALYSIS OF VARIANCE

Source	df	Sum of Sqs. (Corrected)	Mean Sqs.	F	Significance*
Total	26	132	—	--	-----
Temperature	2	5.67	2.84	1.12	-----
Current Dens.	2	1.10	0.55	0.22	-----
Silver Wt.	2	4.36	2.18	0.86	-----
Silver Dens.	2	44.75	22.38	8.85	0.005
Residual	18	45.57	2.53	----	-----

Note: df = degrees of freedom, F = Variance ratio

* Davies: "Design and Analysis of Industrial Experiments, Table C"

F value must exceed 1.70 for significance of 0.1
F value must exceed 3.59 for significance of 0.005

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Table No. 14

Silver Plate Formation Study,
Calculation on Variance

SILVER DENSITY

$$\text{Variance} = \sum X_i^2 - \frac{(\sum X_i)^2}{9}$$

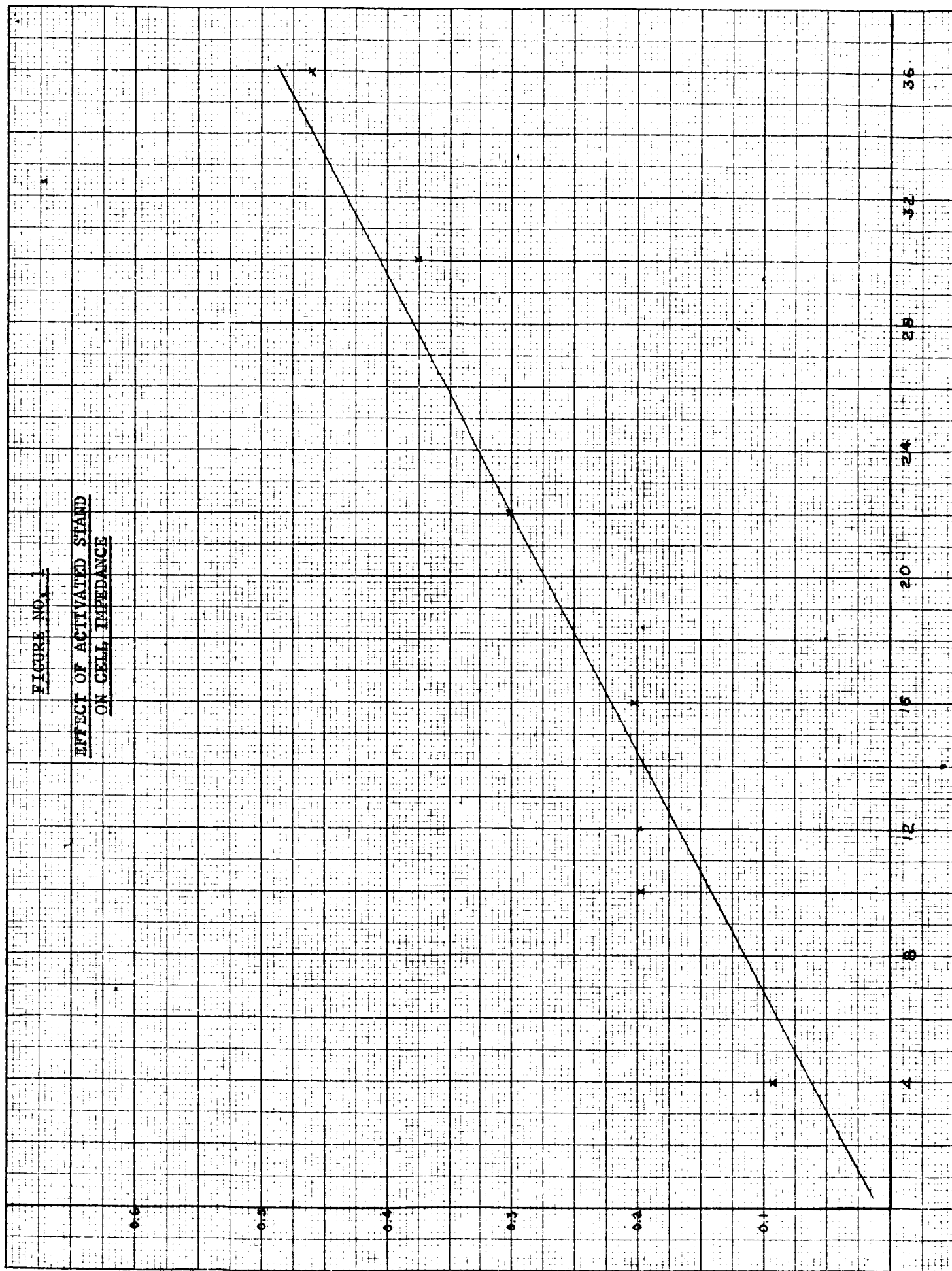Variance (High Dens.) $= 958.57 - (76.72)^2/9$
$= 2.53 \quad (\overline{X} = 8.41)$

Variance (Med. Dens.) $= 903.35 - (89.2)^2/9$
$= 18.88 \quad (\overline{X} = 9.91)$

Variance (Low Dens.) $= 1420.00 - (114.00)^2/9$
$= 16.23 \quad (\overline{X} = 11.57)$

FIGURE NO. 1

EFFECT OF ACTIVATED STAND
ON CELL IMPEDANCE

Cell Impedance (ohms)

Days Activated Stand

31

FIGURE NO. 2

IMPEDANCE OF FRESHLY ACTIVATED CELL

Frequency of Determination (cps)

Cell Impedance (ohms)

32
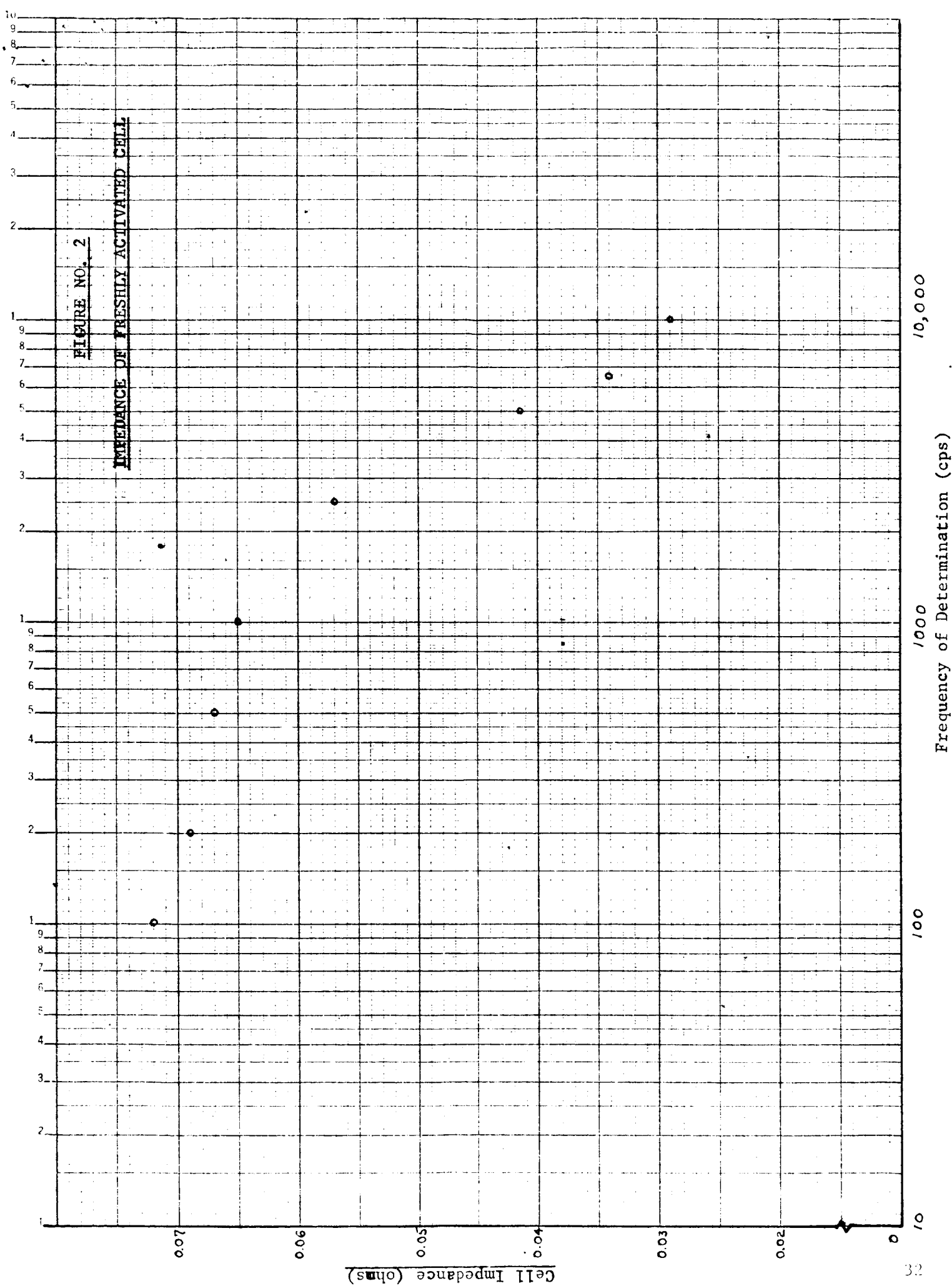
FIGURE NO. 3

CELL IMPEDANCE FOLLOWING THIRTY DAYS
ACTIVATED STAND

(Cell Retaining 1.85 open circuit voltage)

Frequency of Determination (cps)

Cell Impedance (ohms)

FIGURE NO. 4

CELL IMPEDANCE FOLLOWING THIRTY DAYS ACTIVATED STAND

(Cell having 1.60 open-circuit voltage)

Frequency of Determination (cps)

Impedance

$R \times 10^3$ (Ω)

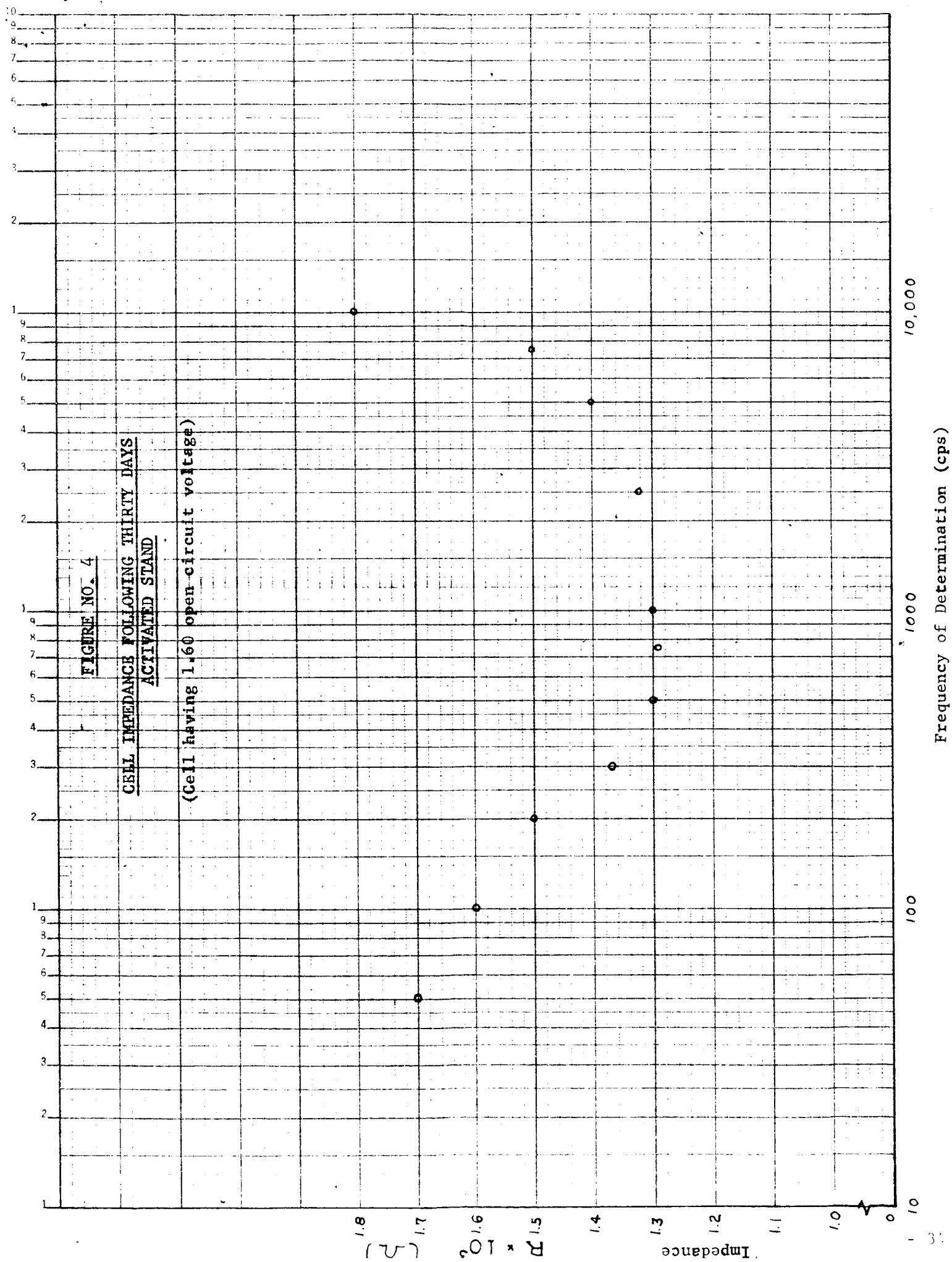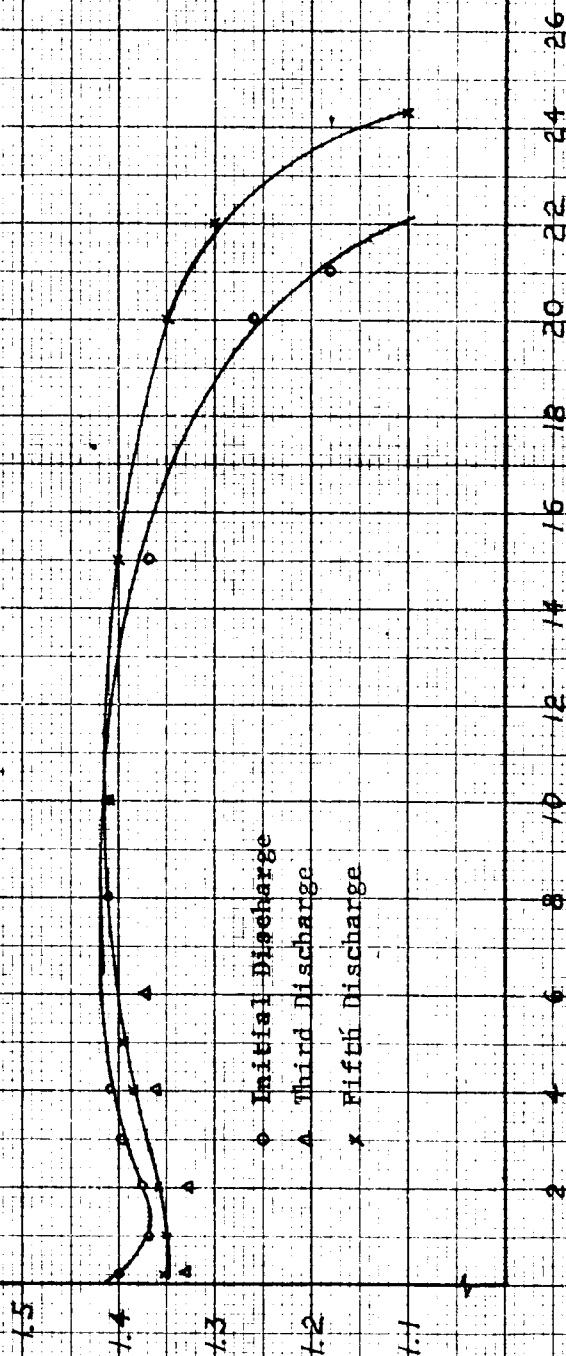FIGURE NO. 5

EFFECT OF STAND ON DISCHARGE CHARACTERISTICS

Separation: 300 Cellophane (2)
#9526 Nylon (1)
R-35-D (1)

⊙ Initial Discharge
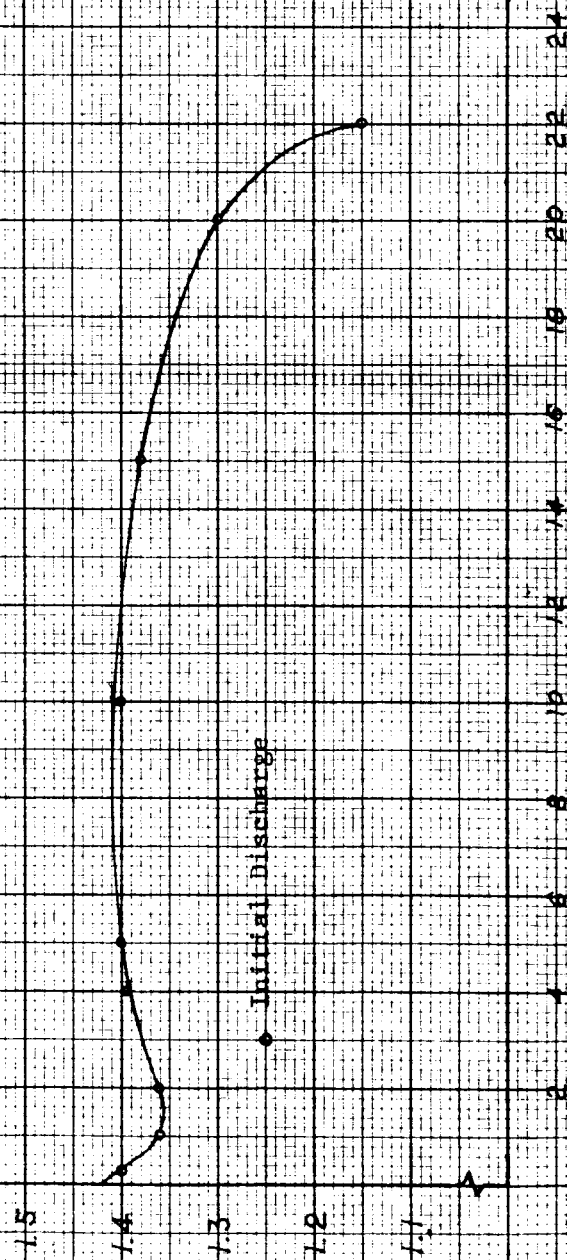▲ Third Discharge
× Fifth Discharge

Discharge Voltage

Minutes on Discharge at Thirty Amperes

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EFFECT OF STAND ON DISCHARGE CHARACTERISTICS

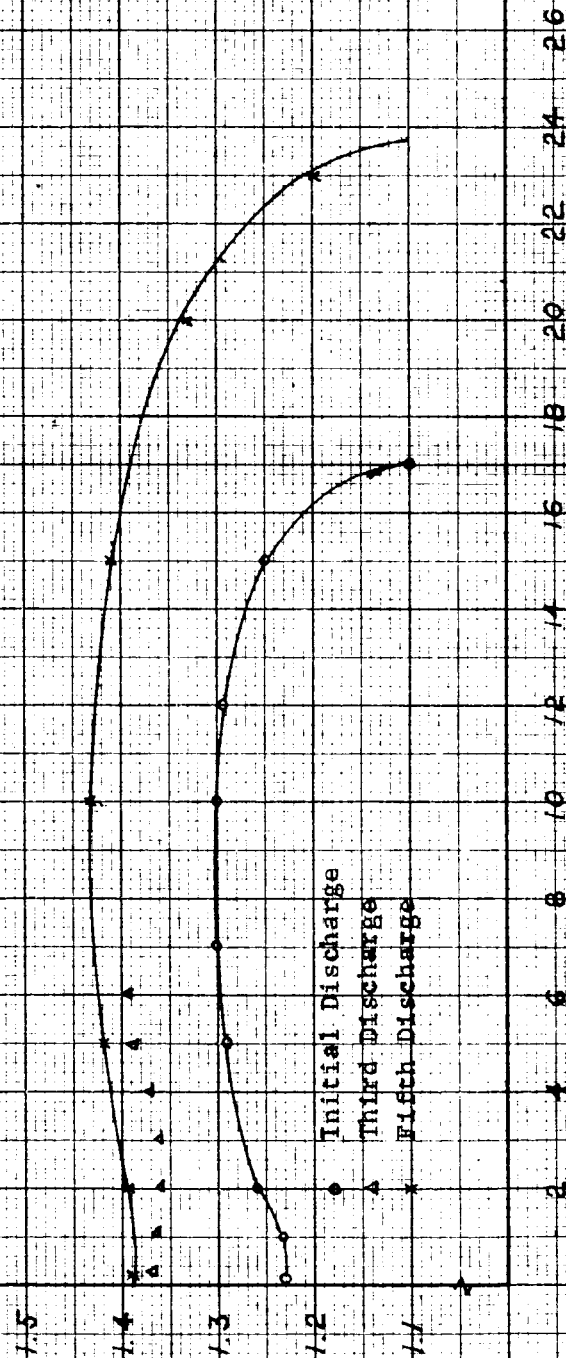Separation: Irradiated Teflon (2)
#5216 Nylon (1)
R-35-D Viskon (1)

Initial Discharge

Cell Voltage

.5 .4 .3 .2 .1

2 4 6 8 10 12 14 16 18 20 22 24

Minutes on Discharge at Thirty Amperes

FIGURE NO. 7

EFFECT OF STAND ON DISCHARGE CHARACTERISTICS

Seperation: 300 Cellophane (2)
EM-470 (2)

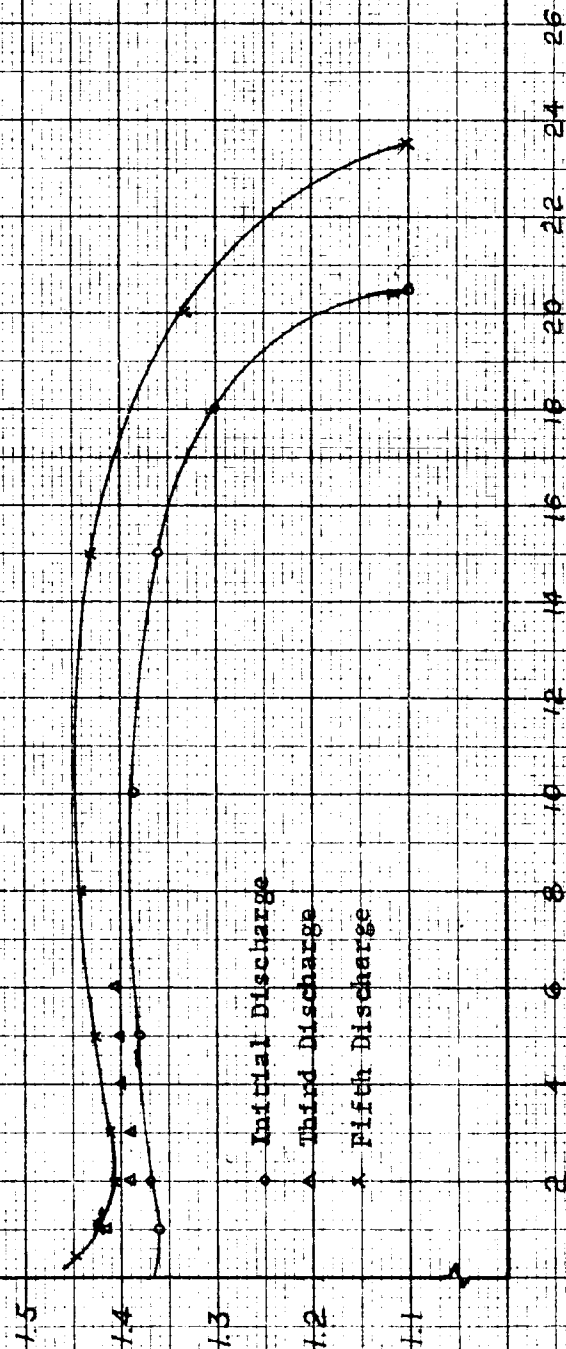Initial Discharge
Third Discharge
Fifth Discharge

Discharge Voltage

Minutes on Discharge at Thirty Amperes

FIGURE NO. 8

EFFECT OF STAND ON DISCHARGE CHARACTERISTICS

Separation: 300 Cellophane (2)
EM-312 (2)

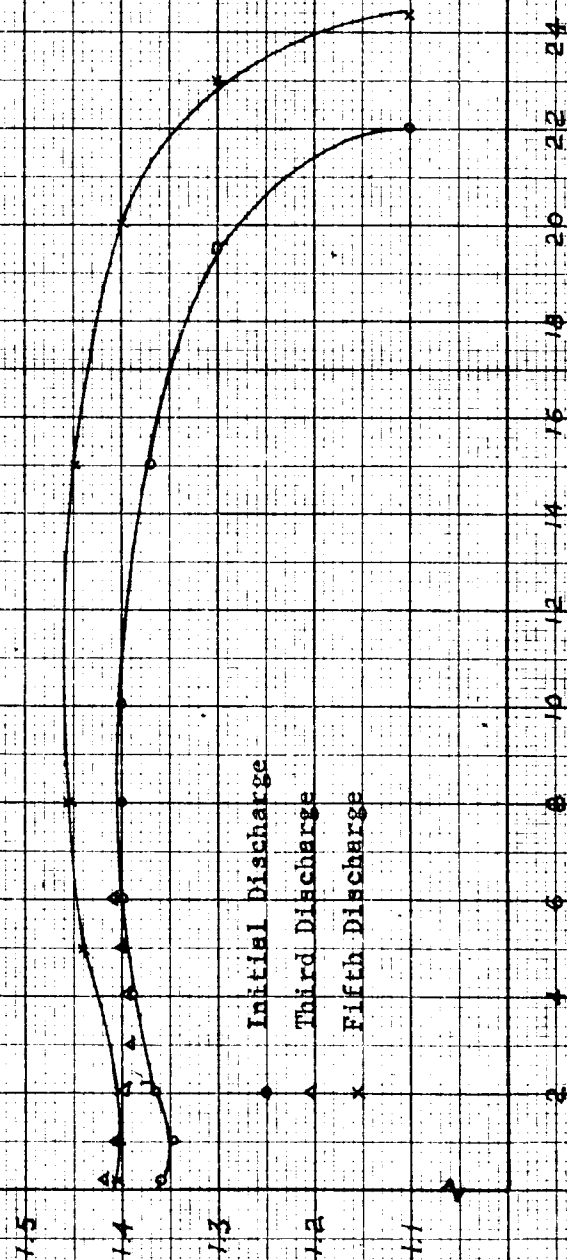Initial Discharge
Third Discharge
Fifth Discharge

Minutes on Discharge at Thirty Amperes

Discharge

FIGURE NO. 9

EFFECT OF STAND ON DISCHARGE CHARACTERISTICS

Separation: 300 Cellophane (2)
M-1406 (2)

Initial Discharge
Third Discharge
Fifth Discharge

Discharge Voltage

Minutes on Discharge at Thirty Amperes

FIGURE NO. 10

INITIAL DISCHARGE CHARACTERISTICS

Separation: 300 Cellophane (2)
EM-317 (1)
M-1406 (1)

Discharge Voltage
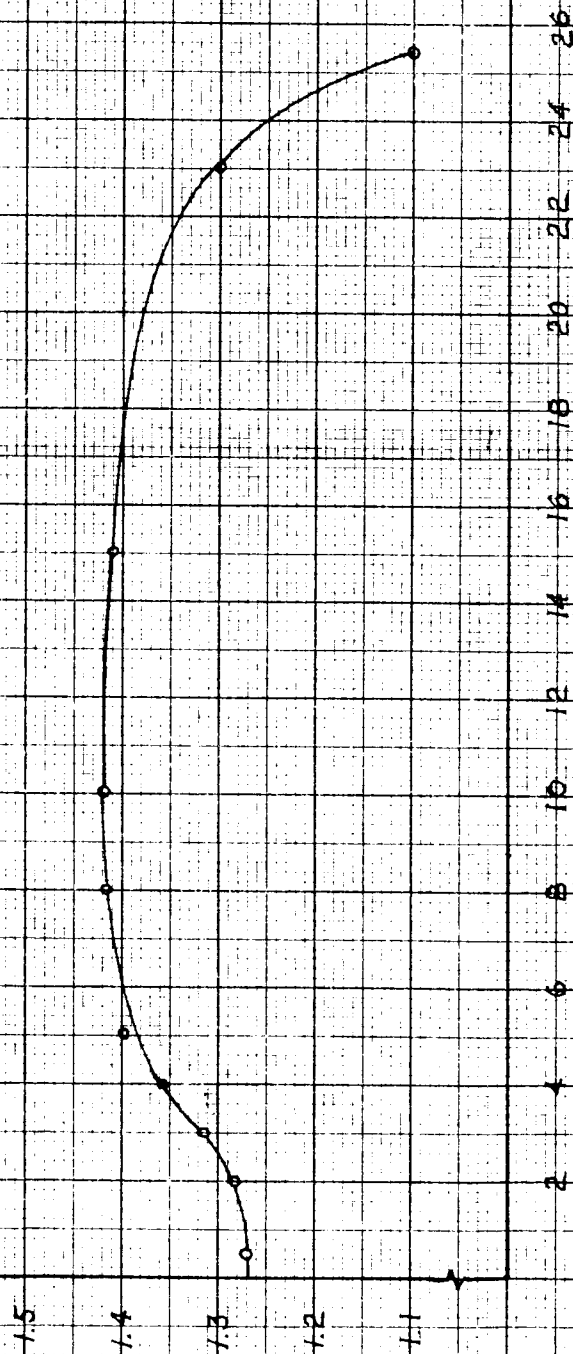
Minutes on Discharge at Thirty Amperes

FIGURE NO. 11

INITIAL DISCHARGE CHARACTERISTICS

Separation: 133 Visking (1)
#9526 Nylon (1)
M-1406 (1)

Minutes on Discharge at Thirty Amperes

Discharge Voltage

FIGURE NO. 12

INITIAL DISCHARGE CHARACTERISTICS

Separation: 300 Cellophane (1)
0.0012 Visking (1)
#9526 Nylon (1)
M-1406 (1)

Discharge Voltage

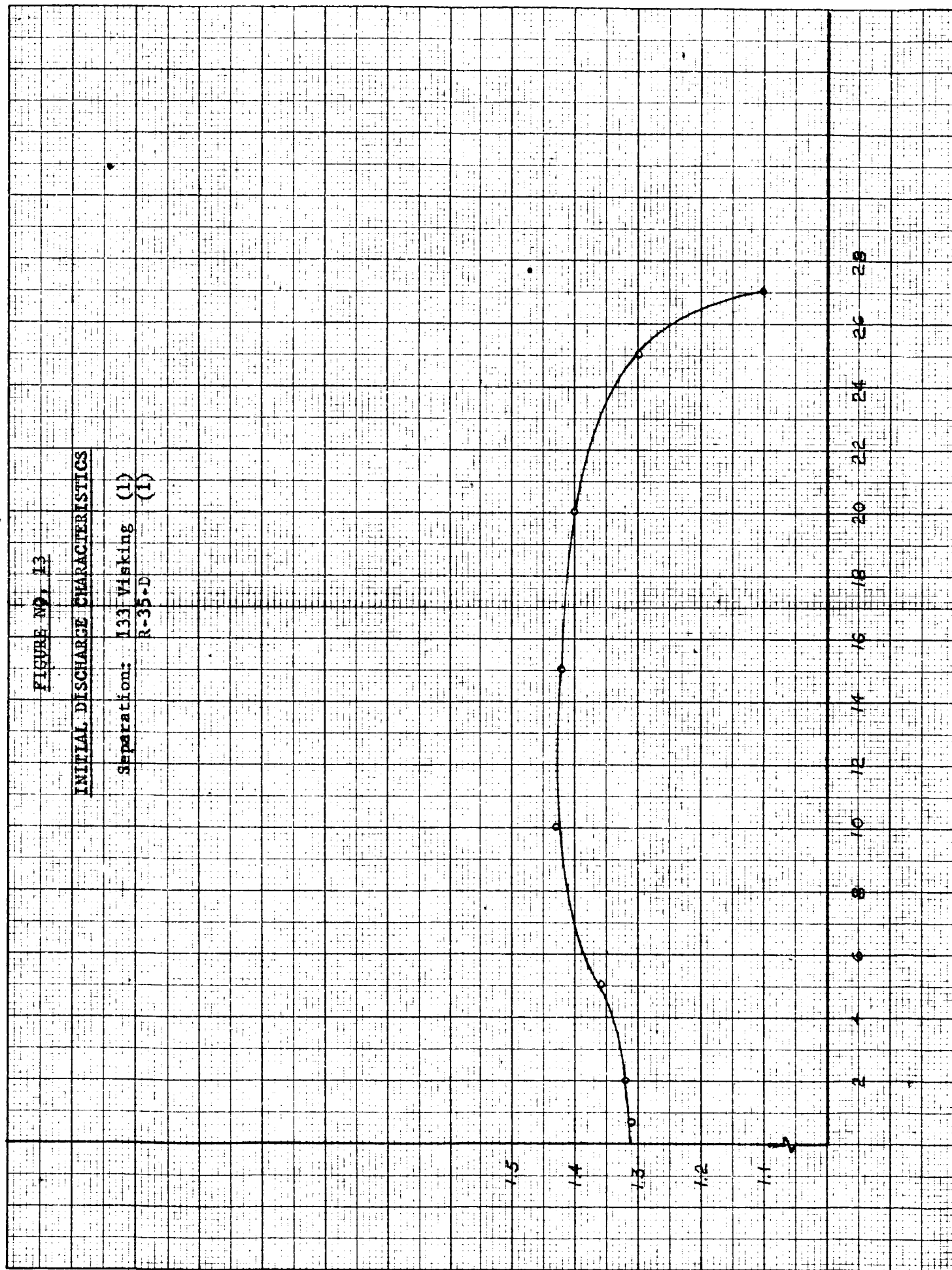Minutes on Discharge at Thirty Amperes

FIGURE NO, 13

INITIAL DISCHARGE CHARACTERISTICS

Separation: 133 Visking (1)
R-35-D (1)

Discharge Voltage

Minutes on Discharge at Thirty Amperes

KEUFFEL & ESSER CO.

FIGURE NO. 14

EFFECT OF SPONGY ZINC APPARENT DENSITY
UPON DISCHARGE EFFICIENCY

Zinc
Apparent Density
(gm/in³)

35
39
44
50
58

Discharge Current Density, Amps/in²

Per Cent Efficiency

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